Gas Chromatography

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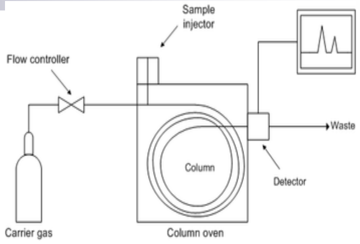
3400
It is strictly forbidden to use this apparatus without instructions from authorized personnel.
Board of department.

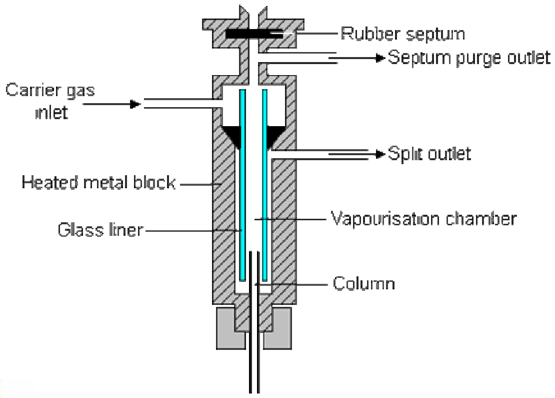
Introduction



- GC - Gas liquid chromatography
  - Separation technique
- In separation techniques (GLC), **THREE** main components are important:
  1. Stationary phase - column
  2. Mobile phase – carrier gas
  3. Mixture of components – vapour pressure (volatility) and solubility.
- **Column:**
  - Stationary phase
  - Thin layer of a liquid stationary phase adhered to the inner wall of the column
- **Carrier gas:**
  - mobile phase
  - is typically helium @ nitrogen
  - flows from a compressed gas cylinder via the regulated pressure at the cylinder
- **Mixture of components:**
  - Concept of **vapour pressure** & **solubility**

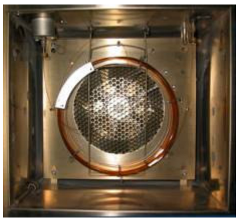
Gas Chromatography

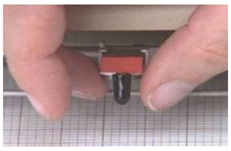
The split / splitless injector



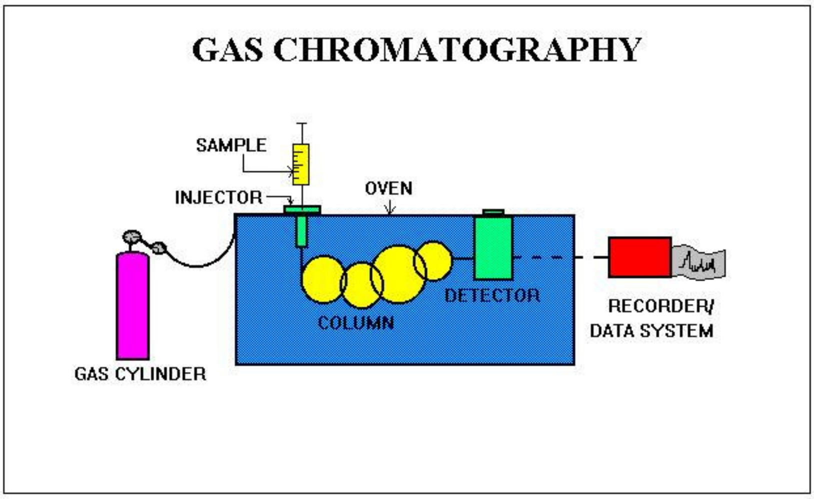
Materials

- Microliter syringe
- Capillary tube
- A tank of He/N gas
  - Depending on the instrument used
- Vaporization chamber
- Oven
- Detector
- Electric/chart recorder
- Integrators (if available)



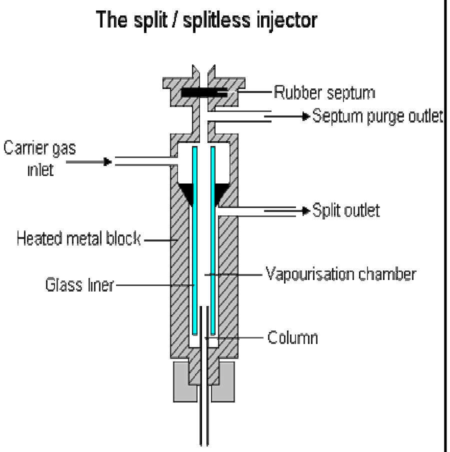


Gas Chromatography Procedures



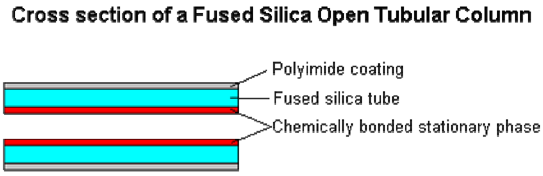
GC Procedure Injector site

- Inject sample
- Control temperatures to vaporize sample
- Mix with carrier gas
- Enters column
- Inlet temperature:
  - Gas samples-since the sample in gas phase, so inlet does not need to be heated. But normally a temp. of 100°C to ensure nothing condenses in it.
  - Liquid sample-require a heated inlet so can vaporise the sample but not too high as can degraded the sample.
    - Start with solvent T


The split / splitless injector

GC Procedure Column-Separation

- Packed columns
  - 1.5 – 10 m long
  - 2-4 mm diameter
  - Glass, stainless steel
  - Solid packing, to max the area min the thickness.
  - High sample capacity
- Capillary columns
  - Typical length 30m
  - 0.1 – 0.5mm inside diameter
  - Coating inside the surface
  - Produce narrow peaks. Allow separation of complex mixtures.
  - Sample size needed normally less than micro litre.


Cross section of a Fused Silica Open Tubular Column

Column Tubing

- Possible tubing material includes:
  - SS-durable, but relatively reactive surface may cause component loss.
  - Glass-fragile, usually required treatment to deactivate the surface
  - Fused silica-used only in capillary columns, inert and robust. The most preferable material to be used.

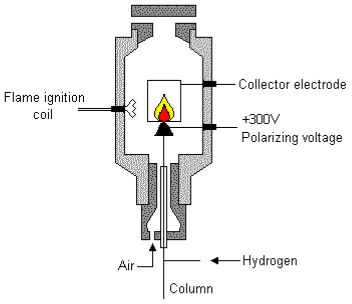
Column Efficiency

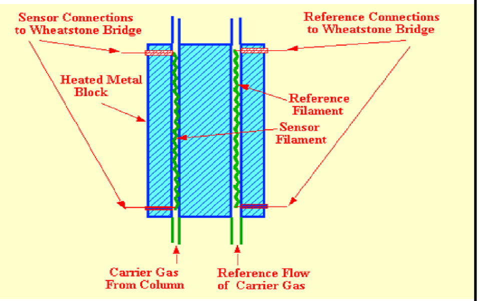
- The column is important to produce narrow, well-separated peaks from multi-component sample.
  - High efficiency-narrow peaks
  - Low efficiency
- Efficiency is determined by the column construction (small tubing diameter and thin stationary phase layer is the best) and the carrier gas flow rate.

GC Procedure- Detector

- Flame ionization detector
  - Destructive
  - H2 and air is mixed with sample
  - Ignited
  - Collector electrode
- Thermal conductivity detector
  - Universal
  - Nondestructive
  - H2 and He is the best selection for the carrier gas due to as the best thermal conductor.
- Electron capture
  - High sensitivity to halogen containing components

Flame Ionizaton Detector



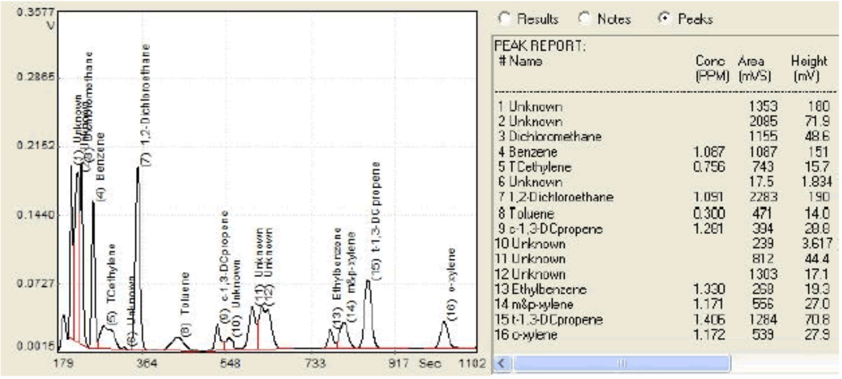

Sensor Connections to Wheatstone Bridge
Reference Connections to Wheatstone Bridge
Heated Metal Block
Reference Filament
Sensor Filament
Carrier Gas From Column
Reference Flow of Carrier Gas

Interpreting Chromatogram

- Peak measurement
  - Time after injection when the peak is detected
  - The size of the peak
- Retention time→ the appearance time is to measure from injection to detection
    - Plumbing time-carrier gas to pass through the column.
    - Retention time-additional time for components interacted with stationary phase in the column.
- Peak size-can be measured either as peak area or peak height, both measured relative to a constructed baseline.
- GC standard pattern can be compared to identify samples that have a high probability of being the same.

Standards for GC

- Calibration – typically preformed with a known volatile compound/mixture
- Attenuation – controlling the height of the peaks.



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PEAK REPORT:
 # Name                     Conc   Area   Height
                            (PPM)  (mVS)  (mV)
------------------------------------------------
 1 Unknown                         1353    180
 2 Unknown                         2085    71.9
 3 Dichloromethane                 1155    48.6
 4 Benzene                  1.087  1087    151
 5 TCethylene               0.756   743    15.7
 6 Unknown                          17.5   1.834
 7 1,2-Dichloroethane       1.091  2283    190
 8 Toluene                  0.300   471    14.0
 9 c-1,3-DCpropene          1.281   394    28.8
10 Unknown                          239    3.617
11 Unknown                          812    44.4
12 Unknown                         1303    17.1
13 Ethylbenzene             1.330   268    19.3
14 m&p-xylene               1.171   556    27.0
15 t-1,3-DCpropene          1.406  1284    70.8
16 o-xylene                 1.172   539    27.9
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Photovac Calibration Run May 22, 2003

Results

- Retention time (R_t)
  - The time required for the compound to pass through the column

$$R_t = \frac{\text{distance from the injection point}}{\text{speed of the chart recorder}}$$

  - R_t is a characteristic value and independent of the presence of any other compound

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Calculate R_t

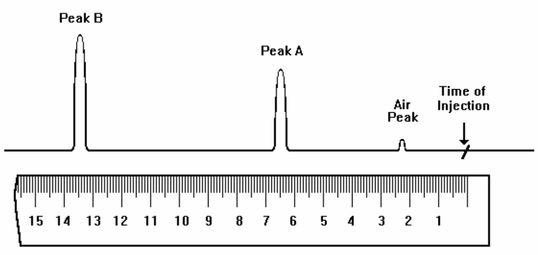
Speed of the chart paper is 2 cm per second

- ☐ Peak A

$$\frac{6.5\,\text{cm}}{2\,\text{cm/s}} = 3.25\,\text{seconds}$$

- ☐ Peak B

$$\frac{13.4\,\text{cm}}{2\,\text{cm/s}} = 6.7\,\text{seconds}$$



Pros for GC

- Speed
- Resolution
- Qualitative analysis
- Quantitative analysis
- Sensitivity
- Simplicity
- Inexpensive

Cons for GC

- Can be slow
- Quantitative analysis
- Destructive
- Volatility